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# BIOFOULING DETECTION AND QUANTIFICATION WITH AN ON-LINE LOCAL THERMAL PULSE ANALYSIS SENSOR

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## 1. INTRODUCTION

In the process industry, fouling and biofouling are considered as complex and misunderstood phenomenon. Control and understanding of the fouling phenomenon is of industrial interest as it leads to reduction in process performances, higher energy consumption, and issues with water management. It is important to have real-time and continuous information about the fouling status of equipment. Biofilm is a consortium of microbial life whose formation occurs in multiple distinct steps (conditioning film formation, initial adhesion, micro-colonies development, macro-colonies included in extra-cellular matrix and detachment). For fouling monitoring, various devices have been reported in the literature including cell counting (coupon), rheological, electrical, chemical, mechanical, optical, sonic, ultrasonic, and thermal methods and exhibiting specificities, advantages, and disadvantages (Fillaudeau, 2007).

In this entry, a fouling sensor (FS) based on local differential thermal analysis was scrutinised and two operating modes, steady (STR) and periodic (PTR) thermal regimes were compared (INRA and Neosens patents). Sensitivity to fouling under steady and periodic heat flux has been scrutinised under abiotic conditions in batch mode at lab scale (Crattelet et al, 2011). In a first step, diffuse (*Pseudomonas aeruginosa*) and dense (*Escherichia coli*) biofilms were generated and monitored with FS sensor in continuous lab-scale bioreactors enabling a control of biofilm growth rate. Sensor limitation and ability to monitor the formation and the elimination (oxidative cleaning) of biofilm were reported. In a second step, the biofilm growth during water treatment of diluted hydrocarbon sludge was investigated.

## 2. METHODS: Local Thermal Pulse Analysis

The concept of the experimental device came from two specific food applications: (i) the use of the hot wire method to monitor milk coagulation in static and isothermal conditions (Hori, 1985, Bellon et al., 1988, Dulac, 1990, Miyawaki et al., 1990, Shimada et al., 1996, Passos et al., 1999 and Auvergne et al., 2002) and (ii) the wall temperature measurement in tubular Joule effect heaters (JEH) to quantify the heat transfer coefficient and fouling mechanism in a continuous heating process. The thermal sensor includes a hot wire, generating a heat flux and two temperature measurements for wall and bulk. One platinum probe acted as a sheathed hot wire sensor. A platinum wire with ceramic and stainless steel sheathes was used, as shown in Fig 1. The ceramic sheath ensured the electrical insulation between the stainless steel and the platinum wire. The platinum wire (hot wire) was connected to a direct current generator (0-50mA). The electric current (I) and potential (V) applied to the standard resistance were recorded and the heat power (P, mW), the flux ( $\phi$ , kW/m<sup>2</sup>) and its electric resistance (R) calculated. The electric resistance enabled us to determine the average temperature of the platinum hot wire, following the relationship (Eq. 1) between T and R for a standard platinum probe ( $R_0$ , A and B are constant).

$$R = R_0 \cdot (1 + \beta \cdot T + \gamma \cdot T^2) \quad (\text{Eq. 1})$$

A thermocouple was stuck half way along the platinum probe and measured the wall temperature ( $T_w$ ) at the sensor – product or deposit interface. The second temperature probe measured the bulk temperature

(T<sub>b</sub>). Each sensor signal (I, V, T<sub>w</sub>, T<sub>b</sub>) was converted using a specific conversion card and recorded on a computer with specific acquisition software (Fillaudeau, 2006).

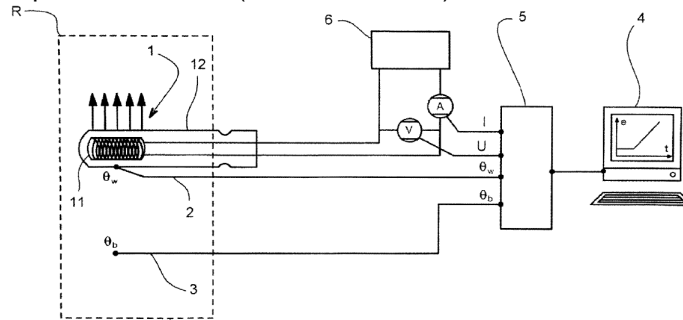


Figure 1: Principle of fouling sensor (INRA patent nFR2885694).

From theoretical standpoint, heat generation inside a sensor with flat or cylindrical shapes could be assumed. In present case, cylindrical sensors are used and heat transfer balance (Eq. 2) is considered in cylindrical coordinates (Eq. 3) with the following equations:

$$\frac{1}{a} \cdot \frac{dT}{dt} = \nabla T + q \quad \text{with} \quad a = \frac{\lambda}{\rho \cdot C} \quad \text{and} \quad \nabla T = \frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \cdot \frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial z^2} + \frac{1}{r} \cdot \frac{\partial^2 T}{\partial \alpha^2} \quad (\text{Eq. 2}) \quad \text{and} \quad (\text{Eq. 3})$$

Geometric and thermal simplifications lead to the simplification of this formula in the platinum wire submitted to the Joule effect (heat generation and heat conduction) and in the sheath (heat conduction) in contact with deposit or fluid. Fouling sensor may be used under various thermal modes (steady and periodic regimes). Considering the sheath surrounding the heat source and in contact with the fluid / deposit, initial and boundary conditions could be formulated in one-dimensional (flat and radial coordinates) analyse, and heat generation equation leads to establish temperature expression versus position and time.

### 3. RESULTS AND DISCUSSIONS

#### 3.1. Biofilm monitoring at lab scale.

First experimental device was a continuous bioreactor fed with a nutrient medium. Nutrient medium was a modified broth biofilm (MBB) containing only one carbohydrate (glucose) whereas biofilm broth (BB) contains a carbohydrate (lactose) and amino acids (yeast extract and casamino acids). Nutrient concentrations are reduced in comparison with a conventional culture medium in order to favour microorganism adhesion. Bioreactor was equipped with two internal loops: (i) a flexible tube in polymer material (Masterflex Tygon 06409-17, D : 8/6,4mm) in which stainless steel strips (304L, S=5.12cm<sup>2</sup>) were immersed (bacterial adhesion count) and (ii) a stainless steel module integrating FS sensor. Overall bioreactor volume is close to 150mL and temperature. Cell broth circulation was controlled with 3 pumps: (i) pump 1 (Masterflex n°7544-01, head: 7017-20) with Q=3 mL/h imposing a growth rate around 0.02h<sup>-1</sup>), (ii) pump 2 and 3 (Masterflex n°7554-85, head: 7017-21) achieving a laminar flow regime around stainless steel strips and a turbulent flow within second loop. Strips colonization by bacteria was measured after 3, 6 et 48h incubation periods by scraping and water rinsing of strip surfaces and then counting after inoculated onto the surface of many agar plates. Fouling sensor was used in steady thermal regime (STR alternately 5min in active mode, P=100mW equivalent to φ=1400W/m<sup>2</sup> and 5min in passive mode, P=0mW) and periodic thermal regime (PTR, 5 cycles at 0.66, 4.5, 7.5, 22.4 and 25.5h, f=0.025Hz, P<sub>max</sub>=100mW equivalent to φ<sub>max</sub>=1400W/m<sup>2</sup>, sinusoidal).

*Pseudomonas aeruginosa* bacteria was inoculated in bioreactor at 8.45 logUFC/mL. Biofilm formation was monitored by counting (strips) : 4.7 log UFC/cm<sup>2</sup> at 3h, 5.2 log UFC/cm<sup>2</sup> at 6h and 7.2 log UFC/cm<sup>2</sup> after 48h corresponding to an average biofilm thickness ranging 100-150µm. Fig 2 indicates that on-line monitoring in STR and PTR do not corroborate cell counting. Thermal deviation of FS remains insignificant. Biofilm generated by *P. aeruginosa* in our operating conditions is supposed to have a "mushroom" structure with bacterial microcolonies enabling a free internal flow. Then colonised (fouled) surface at surface sensor may be inferior to 50% of whole sensitive surface so it is difficult to quantify. Forced convection remains the main heat transfer mechanism at sensor surface. Under these conditions, FS seems inappropriate to monitor biofouling.

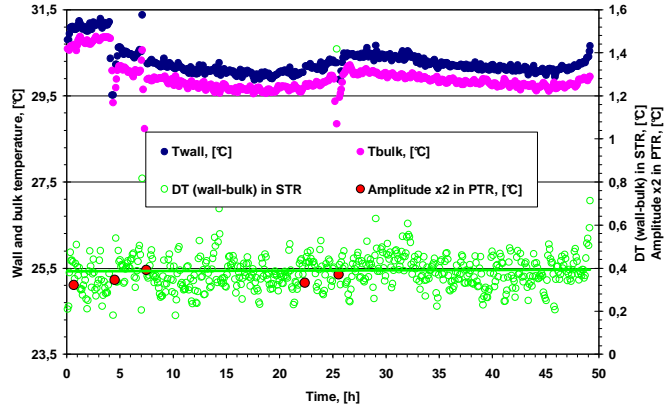
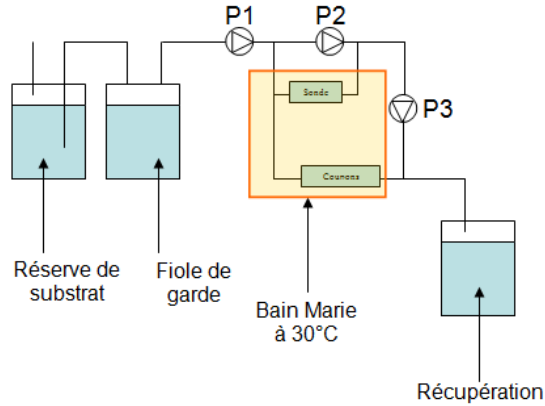


Fig. 2. Experimental set-up based on a continuous bioreactor including two derivation loop (A) and evolution of bulk and wall temperatures,  $T_b$  and  $T_w$ , and wall overheating,  $\Delta T$ .

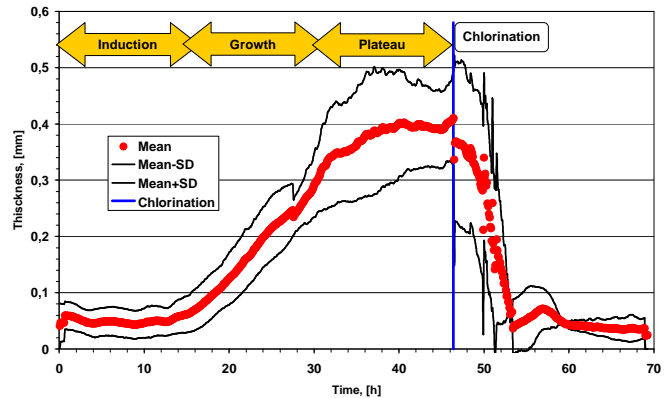
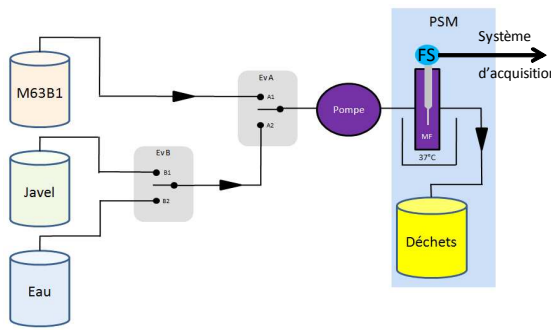


Fig. 3. Experimental set-up including a milli-bioreactor including FS sensor (A) and mean thickness and associated standard deviation during *E. Coli* biofilm formation (steady thermal regime,  $\phi=1600\text{W}/\text{m}^2$ , B)

Second experimental device included feeding tanks and milli-bioreactors (Fig. 3-A). The bioreactors had a cylindrical shape ( $D_{\text{int}}=20\text{mm}$ ,  $H_{\text{liquid}}=200\text{mm}$ ) and were equipped with an immersed tube nozzle (feed) and an overflow. Temperature was regulated with a water bath thermostated at  $37(\pm 1)^\circ\text{C}$ . A soft magnetic stirring insured the homogeneity of broth. Milli-bioreactors were fed alternatively with 3 solutions (nutrient medium M63B1, sodium hypochlorite solution, water) with a peristaltic pump (Watson Marlow 205S,  $Q=1.7\text{mL}/\text{min}$ ). Under these operating conditions, the medium was regenerated every 35min which imposed a growth rate around  $1.71\text{h}^{-1}$ . FS were previously sterilised and was inoculated directly into a well with fresh cell preculture (*E. coli* TG1,  $\text{OD}_{600\text{nm}}=1$ ) before to be plunged into bioreactors. Cell growth and biofilm was generated in optimal conditions and exhibited a dense structure. After growth phase, a plateau phase was obtained (48h) and the feed was switched to sodium hypochlorite solution in order to remove biofilm up to clean conditions.

Under these conditions, rich nutrient medium and weak turbulent flow surrounding the sensors, a dense biofilm is formed and could be observed. From ten experiments and three FS ( $\phi=1600\text{W}/\text{m}^2$ , steady thermal regime), the same tendencies could be observed and confirmed bacterial deposit at sensor surfaces. Initial wall overheating of  $0.4^\circ\text{C}$  (off-set) is observed in agreement with a poor heat transfer coefficient. However this overheating will not affect growth rate and assuming that deposit conductivity  $\lambda=0,6\text{W}/(\text{m}\cdot\text{K})$ , it correspond to  $150\mu\text{m}$  (background signal).

Fig 3-B reports the evolution of mean thickness (and associated standard deviation) as a function of time. Different phases associated with biofilm growth or elimination could be identified:

- 0 to 12h : induction step in which bacteria adhere to sensor surface and adapt to their environmental conditions,

- 12 to 35h : exponential growth phase in which biofilm growth at an average velocity equal to 12 $\mu\text{m}/\text{h}$ ,
- 35h to 48h: stationary or planktonic phase in which a constant thickness of biofilm is observed ( $\approx 400\ \mu\text{m}$ ) and assumed to be a balance between biofilm growth and erosion/removing (shearing),
- after 48h, a chlorination step induces the elimination of biofilm (44 $\mu\text{m}/\text{h}$ ) by a strongly oxidative treatment up to clean conditions (background signal).

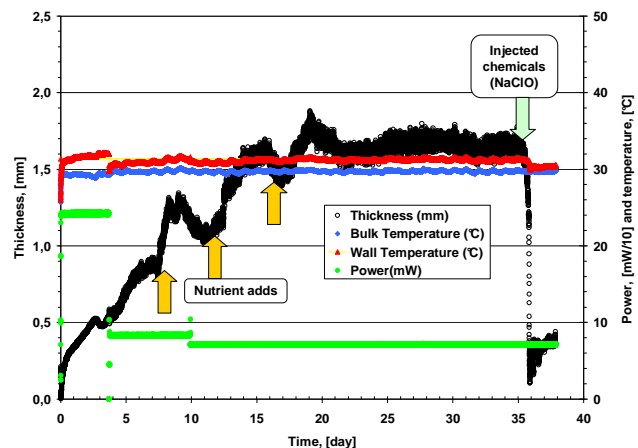
A poor but normal repeatability is noticeable during microorganism cultures in millibioreactor as indicated by standard deviation curves.

### 3.2. Biofouling monitoring at pilot and industrial scales.

Fouling (Biofilm, scale) is a worldwide problem reducing energy efficiency, increasing maintenance costs and creating health risks. The conventional approach to fouling control combines the “blind” application of biocides and anti-scale chemicals with periodic lab testing. This often results in the excessive use of chemicals with the inherent side effects of accelerating system corrosion and increasing toxic waste- not to mention the incremental cost of unnecessary treatments. The wastewater treatment (petrochemical industry, TOTAL CReG, Gonfreville) was investigated in a double jacketed bioreactor ( $V=7\text{L}$ ) equipped with a four blades turbine and including mixing (80-85rpm), pH (pH7.6) and temperature ( $T=30^\circ\text{C}$ ) regulations under aerated conditions (Fig 4-A, Crattelet et al., 2009). A flat FS was used to monitor biofouling thickness. In order to increase FS sensitivity, the dissipated heat power was modulated along bioreaction (240mW between 0 and 3.7days, 83mW between 3.7 and 9.9days and 71mW up to 38 days). FS worked continuously in active mode (2minutes/acquisition). Assuming a thermal conductivity of biofilm around 0.6W/(m.K), on-line thickness of biofouling was estimated. After induction and growth phases (around 10 days), a feeding strategy was associated with FS signal evolution and 3 nutrient adds (100mg/L of oligo-elements and carbon source – hydrocarbon sludge) were realised. Whatever heat flux, the biofilm growth was accurately estimated as reported in Fig 4-B. A significant increase of biofilm thickness is observed after nutrient add. However, none oligo-element nor carbon source can be identified as the limiting factor under investigated conditions. Hydrodynamic limitation of biofilm increase was assumed after 20days (plateau phase). In a final step, a biocide (NaClO) injection was realised in the broth at 35.5 days in order to inactivate microorganism and to eliminate biofouling. A full elimination of biofouling was noticeable including a slight erosion phase followed by a sharp decrease (uprooting).



A



B

Fig. 4. Pilot-plant bioreactor and configuration (A) and evolution of experimental parameters (flux, wall and bulk temperature) and thickness of biofouling under steady thermal regime (B).

## 4. CONCLUSION

A fouling sensor (FS) based on local differential thermal analysis was scrutinised and two operating modes, steady (STR) and periodic (PTR) thermal regimes were investigated. With diffuse (*Pseudomonas aeruginosa*) biofilm, FS do not provide pertinent response. During growth of dense (*Escherichia coli*) biofilms, FS sensor properly detected and quantified biofilm growth and elimination kinetics. Sensor limitation and ability to monitor the formation (dense biofilm, complex biofouling) and the elimination (oxidative cleaning) of biofilm were reported.